MEASURING THE MOISTURE CONTENT OF A VAPOR – GAS MEDIUM WITH A CAPACITIVE PROBE

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A capacitive method is being developed for the measurement of moisture content in a vapor – gas medium. We provide a detailed description of the measuring apparatus, as well as of the method of fabricating and calibrating the probes which are produced on an aluminum oxide base.

The capacitive method of measuring moisture content in a vapor – gas medium – as developed by the authors here – is based on the measurement of the permittivity of the aluminum oxide, which varies as a consequence of the adsorption of water vapor from the ambient medium.

The moisture-sensitive element of the capacitive hygrometer is formed by an aluminum backing to which an aluminum oxide film of the required thickness is applied by means of sulfuric-acid anodizing. The aluminum backing serves as one of the covers of the capacitive hygrometer, with a layer of graphite deposited electrolytically onto the aluminum oxide surface or a thin metal film applied to the aluminum oxide by vaporization in a vacuum serving as the other cover.

An increase in the relative moisture content leads to a drop in the resistance and capacitive refractance of the capacitor, formed by these electrodes.

The equivalent electric circuit of the oxide layer [1] (Fig. 1) is formed of the capacitance C_0 and the resistance R_0 between the aluminum backing and the external electrode. The change in the capacitance C_0 as a function of the relative moisture content is comparatively small, and the resistance R_0 shunting this capacitance is rather large, so that its effect is insignificant. The resistance R_0 is equivalent to the resistance of the pore walls along their length, while R_2 and C_2 represent the resistance and capacitance of that portion of the oxide film which is located between the pore base and the pure aluminum (the so-called barrier layer). With a low relative moisture content the value of R_1 is very high; the shunting effect of C_2 on C_0 is insignificant. Since the adsorption of water by the pore walls with an increase in the relative moisture content increases, the corresponding magnitude of R_1 is substantially reduced and the effective capacitance of the element is determined by the capacitance C_2 of the pore base. Because C_2 can be made several hundred times greater than C_0, the effective capacitance of the element becomes extremely sensitive to variations in the relative moisture content. Therefore, having chosen the film thickness and the density of the porous structure – and these depend on the conditions of anodizing (the nature and concentration of the acid in the electrolyte, temperature, current density, voltage at the electrodes, and duration of the process) – we can achieve a rather sensitive element for the capacitive hygrometer.

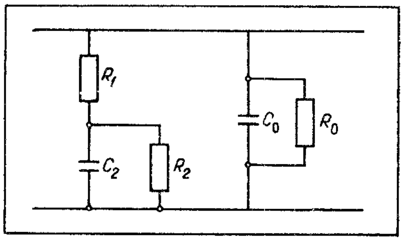

Fig. 1. Equivalent electric circuit of the outside layer.

For the backing material we chose foil of two grades of aluminum (AVO and AVOO) with a thickness of 90-100 μ (40 × 25 mm).

Prior to the anodizing, the foil is polished on a belt wheel, degreased, and electropolished.

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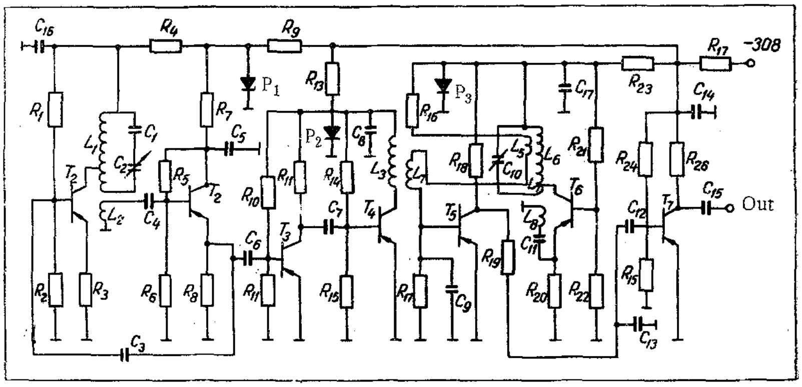

Fig. 2. Measuring circuit for probes of low capacitance, with oxide layer (up to 10 μ).

We tested the most diverse combinations of duration, bath temperature, current density, and kind of current in the annodizing of the aluminum foil in a sulfuric-acid solution. The acid concentration was varied here from 18 to 52%.

To produce outside films with a thickness of 2-3 μ and a maximum porosity, the optimum approach is the following anodizing regime: concentration, 50%; bath temperature, 26-28°C; current density, 25 mA /cm^2; anodizing duration, 25 min; current, alternating.

To produce oxide films with a thickness of 9-10 μ with an elevated porosity, we employ the following anodizing regime: acid concentration, 50%; bath temperature, 18°C; current density, 30 mA/cm^2; anodizing duration, 25 min; current, alternating.

The anodizing process is accomplished with intensive bubbling. The thickness of the oxide film is determined from the voltage breakdown. We know from the literature that the dc voltage on breakdown is equal to 37 V per 1 μ of oxide-film thickness; the pressure of a steel ball on the oxide in this case must be 1-2 kg; the leakage current in this case is equal to 1 mA.

The shape and location of the external electrodes, when coated in a vacuum, is controlled by means of a stencil or a mask cut from a metal strip. Depending on the probe to be fashioned, the edge of the anodized specimen is covered with a 2 mm wide Plexiglas film, on either one or both sides. This serves to fill up the pores of the anodized surface, and this segment thus ceases to be sensitive to changes in moisture content and subsequently serves to maintain contact with the drainage conductor. We used silver applied in a vacuum as the metal coating, sprayed from a molybdenum container at a vacuum of $p = 5 \cdot 10^{-5}$ mm Hg. A liquid-nitrogen trap and a glass wool filter were used to prevent the vapors of the oil from the diffusion pump from entering the working chamber of the installation. From 210 to 230 mg of silver is needed to coat the external electrodes in one spraying operation; the silver is vaporized at a distance of 180 mm between the molybdenum container and the anodized foil. It was found that the thin films produced with a smaller quantity of sprayed silver exhibit extremely high resistance, which has a negative effect on the functioning of the sensing element. On the other hand, the thick films produced with quantities in excess of 230 mg of sprayed silver also reduce the sensitivity of the probe, so that the films become dense and transmit the moisture poorly. We also found that to achieve a rather porous external electrode with a low electrical resistance the process of silver vaporization should be performed with great intensity.

After the coating of the external electrodes, the specimens are immersed into boiling distilled water for 30 sec, which makes the coating more porous and imparts greater sensitivity to the element. However, with longer boiling the coated layer is degraded.

The maintenance of contact between the Al and the drainage conductor, the external electrode, and the other current conductor is accomplished by means of a current-conducting paste.

The use of probes of various sizes and, consequently, various capacitances makes it necessary to design special measuring circuits.

In developing circuits for measurement of small capacitances (up to 500 pF) we made an attempt to satisfy the following requirements rigorously: 1) an absence of a constant voltage component at the sensor; 2) a small amplitude for the variable voltage; 3) a low initial capacitance for the measuring input.

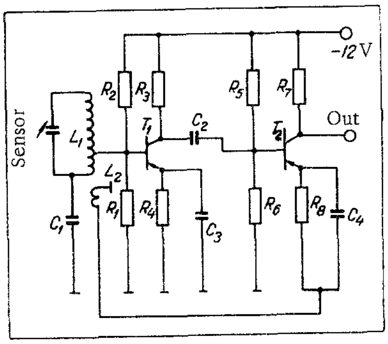

Fig. 3

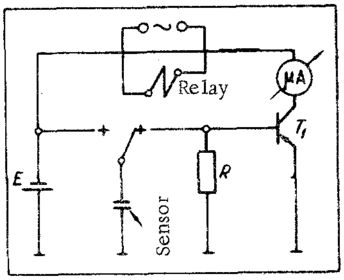

Fig. 4

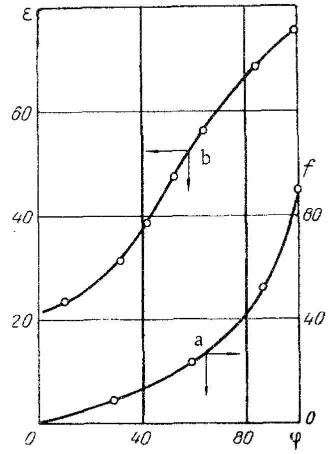

Fig. 5

Fig. 3. Measuring circuit for probes of low capacitance, with a thin oxide layer (2-3 μ).

Fig. 4. Ballistic measuring circuit for probes with substantial capacitance.

Fig. 5. Typical calibration curves for moisture-content probes (ε denotes the scale graduation): a) with low capacitance, using the circuit of Fig. 2; b) with substantial capacitance, using the circuit shown in Fig. 4.

The first requirement is necessary only for moisture-content meters operating with polarizing sensors that are, for example, based on Al_2O_3 in which the dielectric has a large $\tan \delta$. With a constant component for the probe with a large $\tan \delta$ leads to excessive heating of the probe body, which serves to make the readings of the moisture-content meter dependent on the rate of the flow being measured. This same drawback will be encountered when the probe is subject to a substantial variable voltage.

In addition, the amplitude of the variable voltage must be lower than the permissible breakdown voltage of the dielectric. For probes based on aluminum oxide, with $\varphi = 100\%$, this voltage amounts to ~0.6 V/μ [1].

The capacitance of the measuring input leads to a reduction in sensitivity, which can be seen from the equation

$$k = \frac{f_{\max}}{f_{\min}} \sqrt{\frac{C_{100} + C_{\text{in}}}{C_0 + C_{\text{in}}}}. \tag{1}$$

Hence it follows that as $C_{\text{in}} \to \infty$, $k \to 1$, while as $C_{\text{in}} \to 0$, $k \to C_{100}/C_0 \gg 1$. The effect of C_{in} is particularly pronounced in working with probes that are small in size and, consequently, in probes with small C_0.

To satisfy the above-indicated requirements, the authors used circuits in which the probe is hooked into the circuit of a sinusoidal voltage generator.

A change in the moisture content leads to a corresponding change in frequency, which is then measured either with a frequency detector with a electron-tube voltmeter at the output or it is transmitted to the input of the mixer simultaneously with the pedestal frequency, the difference between these two frequencies being measured with a frequency meter at the output of the mixer. Both of these circuits, designed on the basis of these principles, are virtually equivalent in terms of measuring accuracy. They both provide for a measuring accuracy of 1%, which in most cases it higher than the accuracy of the moisture-content probes.

The circuit shown in Fig. 2 consists of generator with transistors T_1 and T_2, and a probe connected into the circuit of this generator, in addition to a buffer amplifier with transistors T_3 and T_4, a mixer T_5, a pedestal-frequency generator T_6, and a low-frequency amplifier T_7. This circuit employs a method for the comparison of two frequencies: the frequency determined by the parameters of the probe and the pedestal frequency. Both of these frequencies are applied to the input of the mixer, and from the input of the mixer a frequency equal to the difference between the two frequencies is applied to the input of the low-frequency amplifier and measured with a ThZ-7 frequency meter. We can thus construct the relationship between the readings of the frequency meter and the capacitance of the probe, which is the same thing as plotting the moisture content of the medium surrounding the probe as a function of the frequency-meter readings. With $\varphi = 0\%$ the generator frequencies coincide (the frequency trim is accomplished with C_{10}) and are equal to 500 kHz. The choice of the frequencies is governed by the operating frequency of the ferrite rings which form the inductances of the circuit. The lowest circuit capacitance (C_0) with a good Q-factor for the circuit was achieved at these frequencies. The stability of the generator frequencies amounts to ±0.1%. The circuit is powered by a stabilized source with a voltage of 30 V. The amplitude of the variable voltage for the circuit amounts to 400 mV.

Another circuit is based on the principle of using a frequency detector to measure the generator frequency, a function of the probe capacitance. This circuit is made up of a two-stage generator, a diode limiter, and a frequency detector at whose output the voltage is measured with an electron-tube voltmeter. As in the previous case, the probe is connected into the generator circuit and measures the resonance frequency of the latter with a change in the moisture content. The outstanding feature of this circuit is the fact that because of the great gain and the stable generation threshold it is possible to achieve a variable voltage in the circuit that is equal to several millivolts. For normal generator operation this voltage must be set in a range of 50-100 mV.

Each of these circuits has a nonlinear scale. It should be noted that these scales can be spread at any point. In the first circuit this is accomplished by varying the frequency of the pedestal-frequency generator; in the second circuit, this is accomplished by altering the tuning of the frequency detector, i.e., by shifting the S curve along the frequency axis through the required amount. Adjustments of this type enable us to determine the zero reading of the recording instrument for any value of moisture content and to use the entire instrument scale for readings ($100\% - \varphi$), switching the instrument to lower measuring limits (a frequency meter in the first case, and a voltmeter in the second case).

The generator circuit shown in Fig. 3, as was the case with the familiar circuit, enables us to achieve a voltage of several score millivolts in the circuit and, consequently, for the probe. This is extremely important when working with probes with a sensitive Al_2O_3 layer of several microns, since it virtually eliminates the likelihood of electrical breakdown of the sensitive layer. As we can see from Fig. 3, the generator circuit is simple. It consists of a two-stage amplifier with transistors, and it operates with a common emitter. The LC circuit is connected to the amplifier input. Generator excitation and voltage control in the circuit are accomplished with the winding of the coupler connected to the emitter of the last stage. For the circuit capacitance C we use the capacitance of the probe. The frequency is measured either with a frequency detector using a voltmeter, or with a frequency meter.

Figure 4 shows the circuit which is best used in working with probes of substantial capacitance (up to 1 mF) to avoid the effect of the relay-contact and installation capacitances which, as indicated earlier, reduce the actually attainable sensitivity.

The operating principle of this circuit is based on the following. A variable voltage from the generator is applied to the relay winding. The reed of the relay switches the probe from battery charging to transistor-base discharge at the generator frequency. The charging and discharging currents are proportional to the probe capacitance which varies with a change in the moisture content. We can thus plot the function $I_{disch} = f(\varphi)$ and measure the discharge current I. The discharge current is applied to the transistor base, intensified, and measured with a macroammeter. In these measurements the transistor must be placed into thawing ice, since it operates on a low current and under normal conditions will be markedly subject to the destabilizing effect of the temperature of the ambient medium.

We use 2 methods to test the sensitivity of the elements and their scales: a) in a hygrochamber, in which it is possible to achieve a relative moisture content of $\varphi = 30$-95% at room temperature; and b) in closed vessels with saturated salt solutions, the moisture content above whose surfaces is well known.

The first measurements were carried out in the hygrochamber. For the measuring surface we used the familiar circuit of 2 voltmeters with a ZG-10 current source. With this circuit we determined the resistance of the probe as a function of the moisture content of the air at various magnitudes of the supply voltage and the ballast resistance. However, this method was not completely successful, since it resulted in a rather large hysteresis as the moisture content of the air varied over wide limits. In this connection, we use a method to measure the capacitance of the probe. The current–voltage characteristic of the probe is a straight line which starts out from the coordinate origin; the slope of the straight line in this case depends on the moisture content for which the readings were taken.

The capacitance of the probe was measured by a method involving the shifting of 2 frequencies, and with the ballistic method considered above.

The circuit based on the shifting of 2 frequencies was used for the probes intended for the measurement of moisture content in the boundary layer of a plate in the presence of heat and mass transfer, whereas the ballistic circuit is intended for probes designed to measure moisture content in electromagnetic dryers. Typical calibration curves are shown in Fig. 5. It should be noted that in this case a small hysteresis is observed for relative moisture content in excess of 85%. The reaction rate of the probe to a pronounced change in the relative moisture content is quite satisfactory. Thus, when removing the probe from a medium with $\varphi = 100\%$ to a medium with $\varphi = 10\%$, within 1 min the probe will correctly indicate the moisture content, i.e., the transition lag of the probe is 1 min (measurement by the ballistic method). The delay time for the probes with an outside-layer thickness of 2-3 μ is substantially smaller and amounts to several seconds. The overall error in the hygrometer is estimated at ±2% of the relative moisture content. With a temperature change from 0 to 80°C we have an additional error of the order of ±5%, in which connection we require additional calibration for the higher temperatures. The stability of the readings given up by the moisture-content probes was checked over a period of two weeks through daily determination of the calibration curve. The maximum differences in the readings, based on calibration with respect to reference points, did not exceed ±0.5%.

A moisture-content probe is geometrically small in size, and it is without any temperature and moisture self-fields. The velocity of the vapor–gas medium exerts no significant effect on the readings of the probe, and it will not break down should moisture penetrate or if there is condensation of moisture on the surface of the probe.

NOTATION

k is the conditional sensitivity of the moisture-content meter;
C_{100} is the capacitance of the probe when $\varphi = 100\%$;
C_0 is the capacitance of the probe when $\varphi = 0\%$;
C_{in} is the capacitance of the measuring input.

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